



## Technical News Bulletin

### Spectrographic Standard Samples of Tool Steels

**S**PECTROGRAPHIC STANDARD SAMPLES of high-speed tool steels are now available from the National Bureau of Standards. Six different tool-steel standards, carefully analyzed and certified as to composition, have been added to the list of over 500 standard samples which the Bureau distributes to analytical and research laboratories for use in controlling chemical processes and maintaining the accuracy of equipment. Designed for calibrating and checking spectrographic methods of analysis, the new standards are complex alloys of iron, chromium, vanadium, molybdenum, tungsten, and cobalt, together with small amounts of other elements.

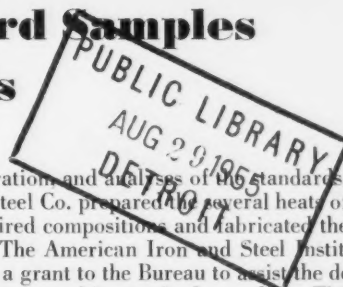
Since the early part of World War II, spectrographic analysis has been extensively applied to the analysis of ordinary steel and nonferrous alloys. Only recently, however, have spectrographic methods been sufficiently developed to permit analysis of complex alloys such as the tool steels used in high-speed machining of metals. Anticipating the application of spectrographic methods to high-alloy steels and the consequent need for standard samples, the Bureau began planning for the production of standard samples of the steel alloys several years ago. Industry cooperated closely with the Bureau, making important contributions in the plan-

ning, preparation and analyses of the standards. The Bethlehem Steel Co. prepared several heats of metal with the desired compositions and fabricated the metal into rods. The American Iron and Steel Institute recently made a grant to the Bureau to assist the development of spectrographic standard samples. This fund facilitated the completion of testing and analyses of the new tool steel standards.

The tool-steel samples are in two forms: rod  $\frac{7}{32}$  inch in diameter and 4 inches long, and rod  $\frac{1}{2}$  inch in diameter and 2 inches long. The surfaces of the rods are finished by centerless grinding. Both forms serve as electrodes for spark excitation in spectrographic analysis.

The rods have a uniform fine-grain structure which results from hot-rolling and annealing of the cast metal. In this condition they can be applied directly to the analysis of molybdenum and tungsten high-speed tool steels prepared in a similar manner and in similar shapes. In applying the samples to the analysis of tool steels prepared by other means or in other shapes, care must be taken to evaluate any possible effects of differences in physical condition on the analyses.

The homogeneity of the rods, both in cross section and along their lengths, was studied by spectroscopic





Homogeneity of new spectrographic standard samples of tool steel being measured in the Bureau's spectrochemistry laboratory. Sample rods are placed in a sensitive photoelectric spectrometer (right), where the alloy's spectrum is measured automatically. Dials at left indicate concentrations of elements present in each sample. Results of many such determinations are examined statistically to ensure that the standard is sufficiently homogeneous before it is analyzed chemically.

and metallographic methods at the Bureau. The samples were found to be of suitable homogeneity for the elements for which concentrations are certified. Chemical analyses of representative rods were made by Bethlehem Steel Co. (Bethlehem Works), Cleveland Twist Drill Co., Crucible Steel Co. (Halcob Works), Carpenter Steel Co., and the Bureau.

The tool-steel standards may be obtained from NBS for a fee of \$8.00 each. A provisional certificate supplied with the standards gives average values of the analyses by the cooperating laboratories. The compositions and sizes of the standards are given in table 1.

TABLE 1. Compositions of NBS spectrographic standard samples of tool steels

Sample Nos. <sup>a</sup>	Mn	Si	Cu	Cr	V	Mo	W	Co
436	836	0.21	0.32	0.075	6.02	0.63	2.80	9.7
437	837	.48	.53	---	7.82	3.04	1.50	2.8
438	838	.20	.17	.17	4.66	1.17	8.26	1.7
439	839	.18	.21	.12	2.72	1.50	4.61	5.7
440	840	.15	.14	.059	2.12	2.11	0.070	13.0
441	841	.27	.16	.072	4.20	1.13	.84	18.5

<sup>a</sup> Sizes: 400 series, rods 7/32 in. diam and 4 in. long; 800 series, rods 1/2 in. diam and 2 in. long.

## Determination of Color Differences

ACCURATE MATCHING of colors is becoming increasingly important to modern industry. Household appliances such as stoves, refrigerators, washing machines, and clothes dryers may be assembled from parts produced on different production lines and finished with different materials. Noticeable color differences between parts may be objectionable to the customer and may hurt the sale of the product. Likewise, for many other uses, such as advertising signs, trademarks, traffic lights, and railroad signals, color is rigidly specified and closely controlled.

For many years manufacturers and purchasers of colored materials have employed inspectors to determine whether or not two samples are sufficiently similar in color to be called a "match." Frequently inspectors cannot agree, and in recent decades instruments have been developed to measure color and color differences precisely. But the extent to which instrumental values of color differences agree with estimates of human observers is a question that has never been satisfactorily resolved.

To obtain data which may lead to a solution of this problem, the Bureau, in cooperation with the Porcelain Enamel Institute, embarked some years ago on a long-range research program. Although much experimental work remains to be done, preliminary phases of the investigation have provided significant information on the evaluation of color differences. This initial stage

of the problem has been carried out by J. C. Richmond, H. J. Keegan, and H. K. Hammond of the NBS staff and R. S. Hunter, Director of Hunterlab, with the cooperation of a number of industrial laboratories.

For estimation of color differences, approximately 200 porcelain enamel specimens were prepared in 15 groups of 13 panels each. Each group contained one panel which represented the "standard" color. The other panels of the group represented departures of approximately one and two steps in each of the six directions: lighter, darker, stronger, weaker, and toward the two adjacent hues. Thirty-four observers from 10 cooperating laboratories were then asked to estimate visually the relative magnitude and most prominent directions of the color differences between each specimen and the corresponding standard for that group. Nineteen of the 34 observers were classed as experienced in color matching while 15 were without experience in color work.

The data from the two groups of observers were averaged separately and compared. While individual estimates of the color difference between any one specimen, and its standard varied widely, the mean values of color-difference estimates for inexperienced observers were not significantly different from those of experienced observers. The statistical scatter of the estimates by the two groups of observers was also about the same. In 9 of the 15 sets of specimens, the differ-



In measurement of color differences, color specimens (right) are placed successively at the top of the colorimeter. When galvanometer (left) is balanced, instrument dials read numbers from which color differences are computed. A current study by NBS and the Porcelain Enamel Institute indicates that color-difference meters are likely to be more reliable in determining color differences than are human observers.

ence between the statistical scatters, expressed as the average of the geometric standard deviations, was less than 5 percent. In the other six groups, the inexperienced observers had the highest scatter in three colors—brown, gray, and purple—and the experienced observers had the highest scatter in the three others—fawn, light blue, and black.

In general, there was good correlation between experienced and inexperienced observers as regards the size of the statistical scatter for the various colors. Both groups tended to have smaller scatter for the colors near middle gray in the three dimensional color diagram—such as buff, fawn, dark green, gray, light blue, and cream—and larger scatter for the more saturated colors—such as purple, black, orange, brown, red, and white. A possible explanation for these trends might be the background against which the specimens were placed for viewing. Although the color of the background was not specified, it is very likely that in every case it was closer to middle gray than to the saturated colors.

The normal human eye is very sensitive to color differences and can tell quite easily when two colors match. However, it is much more difficult for the eye to judge the magnitude of departure from a color match. Colorimeters, on the other hand, are readily provided with numerical color scales from which differences may be precisely obtained. However, it is difficult to provide colorimeters with response functions that duplicate exactly those of the eye. Also, the magnitude and direction of color differences computed from instru-

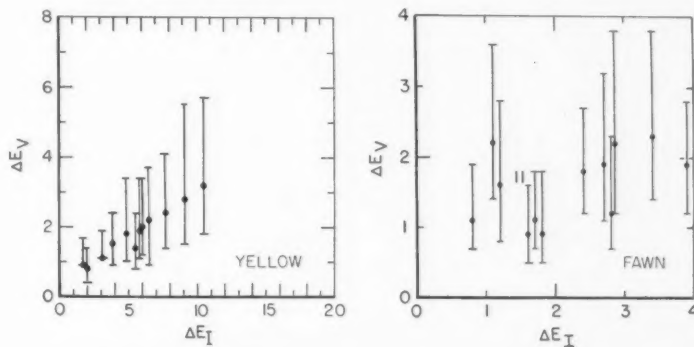
mental data are functions of the color difference equation used.

The present investigation seeks to determine the extent to which colorimeters now in use can evaluate color differences and color-difference components in a manner which correlates with human vision. As a first step, the visual estimates of color difference were plotted against the corresponding instrumental values. Separate graphs were plotted for the three components of color differences: hue, lightness, and saturation. For some colors, the correlation between visual estimates and instrumental measurements was quite good, for other colors there was no apparent correlation, and in a few cases there was a suggestion of negative correlation. The most likely explanation for the cases of poor correlation is simply that the observers were given too difficult a task. That is, the color differences were so small that the observers could not agree on the magnitudes or directions of the hue, saturation, or lightness components of the differences.

While the extent to which instrumental measurements of color difference correlate with visual estimates has not been completely determined, useful conclusions can be drawn from the results obtained thus far. In general, it appears that color-difference meters properly operated are likely to be more reliable than the estimates of a single observer or even a small group of observers. Correlation of instrumental measurements with visual estimates will probably be satisfactory in most cases but should be verified for each color where such correlation is important. However, when color differences are less than two NBS units, low correlation may be expected. The numerical size of an instrumentally measured color difference will depend on the instrument and the color-difference equation used.

The investigation of color difference measurement is continuing. Present plans call for measurement of the

Visual estimates of color differences ( $\Delta E_V$ ) plotted against corresponding instrumental values ( $\Delta E_I$ ). Correlation between visual estimates and instrumental values is quite good for yellow (left) but rather low for fawn (right). Spread of visual estimates for each specimen is indicated by length of the vertical line.



porcelain enamel specimens with other types of colorimeters. These data, together with those from other sources, should ultimately lead to the development of a simpler and generally more useful equation for computing color difference. The spectrophotometer will be used to compile spectrophotometric data for each specimen, and these data will also be used to compute

the magnitude of color differences between specimens. Although the spectrophotometer is the fundamental color measuring instrument, until recently it had seldom been used to measure small color differences. Development of analog computers to automatically integrate spectrophotometric curves has now made this use feasible.

## Electronic Voltage Regulators

### Separate Screen Supply

### Simplifies Circuit Design

**R**ELATIVELY SIMPLE, highly stabilized voltage regulators have been designed by Sherwin Rubin of NBS by adding a separate screen supply to beam power series tubes. In these regulators, tubes having low ratios of screen-to-plate current are combined with screen grid supplies using semiconductor rectifiers. As a result, the series tubes of these circuits have slightly lower efficiency but much higher amplification factors than the low- $\mu$  triodes usually used in voltage regulators. Several units can be interconnected to supply a number of voltages to a piece of equipment. In this case, the circuit can be simplified by energizing the screen grid of a lower-voltage regulator from the output of a higher-voltage regulator.

A voltage regulator usually consists of a series tube, a d-c amplifier, and a source of reference voltage. It is commonly used in conjunction with an unregulated voltage source to deliver a regulated voltage output. Acting as a "variable resistor" connected between an unregulated source and a load, the series tube is controlled by the d-c amplifier. Signals representing the difference between a portion of a load voltage and a reference source are fed to the input of the d-c amplifier, and the amplifier output voltage is used to control the "resistance" of the series tube. For every change in load voltage caused by a change in either line voltage or load current, a corrective voltage is applied to the control-grid of the series tube, resulting in an essentially constant voltage across the load.

Although it is desirable to have a series tube that combines high efficiency with high amplification, an increase in one of these factors can in general be achieved only at the cost of a decrease in the other. Low- $\mu$  triodes such as the type 6AS7 are very efficient because, under conditions of zero control grid bias, they can pass a large amount of current with a small plate-cathode voltage drop. On the other hand, a triode-connected (screen grid tied to cathode through a small resistance) beam power pentode has a higher ampli-

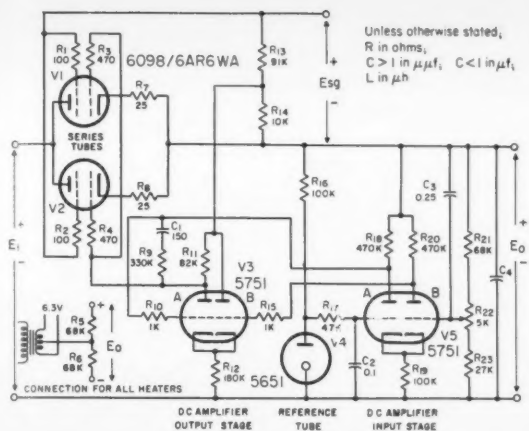


Power unit containing four regulated voltage sources has outputs of +300 v, +150 v, -150 v, and -300 v. All regulators use pentode-connected beam power tubes, and the output of any unit may be varied  $\pm 10$  percent by means of switches along the bottom of the front panel.

fication factor but requires a larger plate-cathode voltage drop for the same current and the same grid bias conditions. The addition of a separate screen voltage source provides a unit that has a higher effective amplification factor than a triode-connected beam power tube. The efficiency of the regulator approaches that of a low- $\mu$  triode.

A 300-v regulated power supply was constructed at NBS using pentode-connected 6098/6AR6 series tubes and a selenium bridge rectifier-RC filter type unregulated screen supply. The regulator contains a two-stage d-c amplifier with a type 5751 twin-triode differential amplifier in each stage. Laboratory investigations show that the output voltage has a ripple of 1 mv when the input-voltage ripple is 30 v and the screen-voltage ripple is 1 v. If the same regulator has an input-voltage ripple of 1 v and a screen-voltage ripple of less than 0.5 v, then a change in line voltage from -10 to +10 percent of nominal value, together with a variation in load from 25 percent of full load to full load, results in an output voltage variation of less than 0.2 v. During this study, the input voltage was 335 v at





Circuit diagram of NBS voltage regulator designed for either positive or negative 300 v output. Screen grid voltage,  $E_{sg}$ , should have a minimum value of 150 v. Tubes of 6CU6/6BQ6 type may be substituted for 6098/6AR6WA shown. For such operation, minimum screen voltage is 90 v. Beam power plates (not shown in schematic) are connected internally.

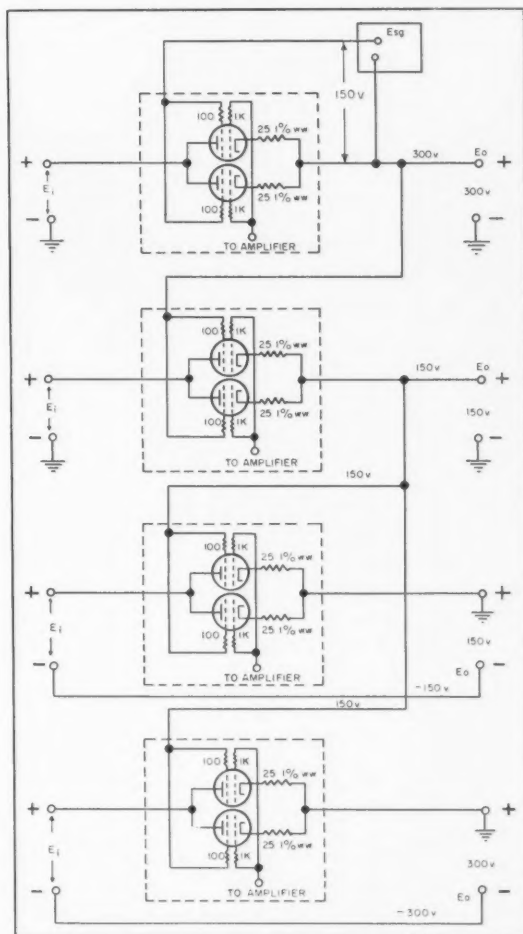
full-load current and at minimum line voltage. The load current was 100 ma per tube. There was a drop of 2.5 v in the cathode equalizing resistors inserted between the cathode of each series tube and the regulated output.

The output impedance of a power supply using the regulator is less than 1 ohm from direct current to over 150 kc. This low impedance is achieved with a single RC feedback network in the d-c amplifier and a 4- $\mu\text{f}$  capacitor across the regulated output. Such simplicity in the d-c amplifier section of the regulator is the result of the high gain of the series tube section. This high gain makes it possible to reduce the number of stages in the d-c amplifier to achieve a given magnitude of loop gain, and also to reduce the plate voltage variation (plate swing) requirements of the output stage of the d-c amplifier. Both factors contribute to the simplification of the interstage coupling and stabilizing networks that would otherwise be required in such a high-gain feedback control system.

If a piece of electronic equipment requiring more than one voltage incorporates several regulators with pentode-connected beam power series tubes, the output of one supply may be used to feed the screen grid of a lower-voltage regulator series-tube and thus eliminate all but one of the separate screen supplies. Such a unit has been constructed at NBS in which the positive 300-v supply feeds the screens of the series tubes in a positive 150-v supply. The positive 150-v supply, in turn energizes the screens in a negative 150-v and a negative 300-v supply. In each case the screen grid to cathode voltage of the tubes was kept at 150 v.

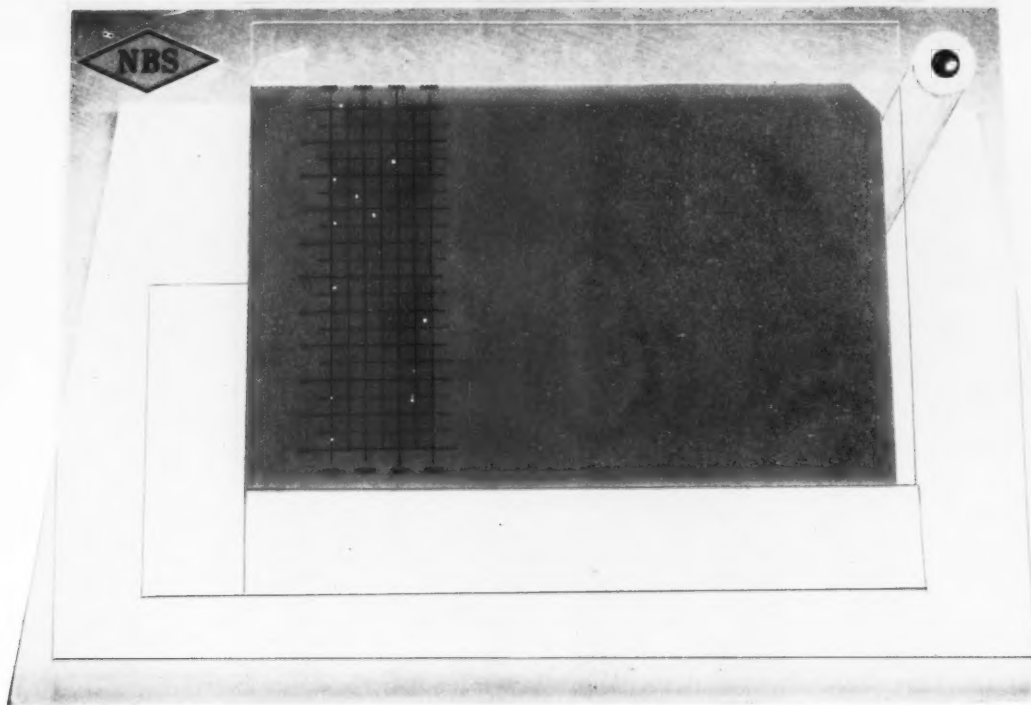
Regulators using the 6CU/6BQ6 type of beam power pentode for the series tube have also been investigated

at the Bureau. These tubes will pass 100 ma of plate current when the plate-cathode voltage is approximately 32 v and the screen-cathode voltage is 90 v. Under these conditions the screen grid will draw approximately 12 ma. When the plate cathode voltage is 100 v, 100 ma of plate current and 7 ma of screen current will flow with a screen-cathode voltage of about 90 v. Under these conditions the plate dissipation is 10 w while the screen dissipation is only 0.63 w. This is typical of the extra dissipation due to the separate screen supply.



Four voltage regulators can be combined in one instrument to provide four separate output voltages. Only one external screen grid supply is required. It is used in conjunction with the +300 v regulator. The +300 v regulator feeds the screens of the +150 v regulator. The +150 v regulator in turn energizes the screens of the -150 v and -300 v supply. This use of regulators themselves to supply voltage to the screen grids of other regulators contributes to the simplification of the equipment. Beam power plates (not shown in schematic) are connected internally.

## Instruments Reference Service



A group of selected index term cards has been placed on the reading illuminator used with the instrument document indexing system. The small spots of light on the overlaid grid indicate the serial numbers of the documents indexed under a particular group of descriptive index terms.

**A**N instrumentation reference service has been established at NBS as a means of providing Government laboratories with more complete access to existing information on measuring instruments, controls, and data handling devices. This service, devised and maintained by the Office of Basic Instrumentation, is designed to increase the effectiveness of scientists working on Government research by helping them to arrive more quickly at the frontier of a particular area of investigation. Economies are also anticipated through elimination of unnecessary duplicate effort. Although the service is presently limited to Government agencies and their contractors, the techniques used should be of interest to others having similar indexing problems.

An improved system for indexing sources of information is used in the reference service, and inexpensive hand-operated indexing equipment has been designed and constructed by Joshua Stern, who directs the reference center. The system is based on the newer concept of indexing which at NBS is referred to as the multidimensional or multiaspect approach.

Scientific research and development projects must be planned and conducted against a background of prior or current work in the same or related fields. The usual method for acquiring this background in-

cludes an extensive survey of the literature. Because of the large number of ideas which relate to any one subject, such a survey is normally time consuming.

In the field of instrumentation, the search problem is especially burdensome because the literature is not so well organized as in more compact fields. Instrumentation information is not adequately covered by abstracting and indexing services whose main interest is in data obtained by use of the instrument. Another problem involved in instrument literature is the absence of suitable, efficient classification systems.

The Bureau realized the need for an instruments documentation system that would minimize the problems of indexing, storing, and searching. Consideration of various methods led to the adoption of a multidimensional approach. This type of system is based on the realization that the same information may be sought from many points of view. Thus, it is designed so that the required information can be found at the common focus of a number of such search viewpoints.

Several considerations led to the selection of a hand-manipulated punched-card method which is the converse of the more conventional punched-card system. This converse method appears to have been conceived some years ago, perhaps independently, in France,<sup>1</sup> Eng-



Hand-operated indexing equipment devised for use with the recently established NBS instrument reference service. A multidimensional indexing system is used because it reduces time required to conduct a document search. Equipment consists of a set of index cards (foreground), a manually operated punch (center), and a plastic reading illuminator (background).

The punch operator inserts separately into the punching machine each of the cards containing the terms chosen to index a given document. The machine positions the card and punches a small hole in it at a position determined by the serial number of the document. Each associated card then has a hole in the same grid position. The cards are returned to the file, the machine is set to the next grid position, and the system is ready for indexing another document. Since the grid pattern provides a total of 18,000 punch positions, one set of index cards can accommodate a collection of 18,000 references.

To carry out a document search, the investigator removes from the file the cards containing the index terms associated with the type of references he is seeking. These cards are placed on a plastic reading illuminator which properly aligns them. Any holes common to all the cards will then be seen as small spots of light. The positions of these visible bright spots identify references common to all the selected cards, which are therefore the references sought.

An example may clarify the search procedure. Suppose a scientist who has devised a method based on electromagnetic induction for measuring the flow of blood in an organism wishes to know of previous work in the same field. He selects from category 1, "measurements," the card for "flow," from category 2, "principle," he selects "electromagnetic" and "induction;" and from category 3, "object," he selects "blood." He places these cards in the register on the illuminator and reads out the position of any holes that appear illuminated. The grid numbers of these holes are the serial numbers of the related documents.

If, on finding that previous work has been done on such devices, he wishes to learn whether any patents have been issued, he selects from category 8, "character of document," the card for "patent." He adds this to the stack previously selected, and notes the remaining holes, if any, which relate to patents.

The application of this system at the NBS Office of Basic Instrumentation has begun with partial coverage of current periodicals supplemented by coverage of abstract journals. Future plans call for an increase in the proportion of direct coverage of current literature.

<sup>1</sup> Classification, classement, rangement et selection, by G. Cordonnier. *Revue Mensuelle de l'Organisation*, April-July 1951 (CNOF, 57 Rue de Babylone, Paris 7e.).

<sup>2</sup> A punched card system of indexing to meet special requirements, by W. A. Batten, *Report of the 22nd Conference, ASLIB* (4 Queens Gate, London, W. 8.) pp. 37-39, 1947; U. S. Patent No. 1,351,692, dated August 31, 1920. Means for compiling tabular and statistical data, by Herbert Edward Soper.

<sup>3</sup> Private communication from Prof. H. A. Toops, Ohio State University, Columbus, Ohio; Specialized files for patent search. Chapter 10: Punched cards, their application to science and industry, edited by R. S. Casey and J. W. Perry (1951).

land,<sup>2</sup> and the United States.<sup>3</sup> Usually in punched-card systems a card represents a document, and the indices assigned to that document are noted by punching holes or notches at appropriate positions on the card. In the converse system used by NBS, a card represents an index term, and the identity of the document to which that term applies is noted by punching a hole at the appropriate location on the card. The location of the hole is determined by the serial number assigned to the document. As applied to instrumentation information, the system comprises a set of index cards, a manually operated punch, and a reading illuminator.

The NBS index system uses 5- by 8-inch cards with a corner clipped from each to permit quick unambiguous orientation. Each card carries an index term in its upper left-hand corner. The cards are filed alphabetically within the following categories, or dimensions (the approximate number of terms in each category is given in the parentheses):

1. Measurand (condition, physical property, quality, quantity, factor or effect to be measured or controlled) -- (255)
2. Principle of operation ----- (289)
3. Object (the material or thing on which observations are being made) ----- (76)
4. Name of instrument, component, or accessory ----- (52)
5. Field of application ----- (21)
6. Stage in instrument development sequence ----- (49)
7. Function in measurement or control ----- (21)
8. Character of document (review, bibliography, patent, Government report) ----- (100)
9. Instrument performance characteristics and ranges ----- (183)
10. Limitations on measurand ----- (183)

Also under consideration is an additional category to provide an author index that is coded in terms of the first few letters of the author's name.

The card file can be expanded to include new terms as desired. Terms which are found to be ineffective can be eliminated at will. This flexibility is an important advantage of the system.

An integral part of the index is a catalog of terms appropriate to each category. In operation, a classifier selects those descriptive index terms which most aptly indicate the information contained in the document to be indexed. No restrictions are placed on the total number of such terms which may be associated with a single reference.

## Improved Method for Applying Cermets



Cermet coatings—combinations of heat-resistant metals and ceramics—are used to protect metal parts against oxidation at high temperatures. Recently the Bureau developed a cermet coating, known as M-60, that can be applied to a basis metal as easily and rapidly as an ordinary ceramic coating. The process employs the usual ceramic coating procedures to replace the former flame-spraying method. In the first step, the coating slip is prepared by grinding the chromium-boron-nickel cermet powder in a ball mill with a ceramic frit and a small amount of enameling clay and water until the particles are as fine as required for the particular application.

**A** RAPID, economical process for applying a common type of cermet coating to a metal base is now in use at the National Bureau of Standards. The cermet powder in this case is a chromium-boron-nickel mixture that is combined with a ceramic frit. The process employs ordinary ceramic coating procedures to replace the former flame-spraying method. Providing a hard, high-temperature-resistant facing for metal parts, the coatings have excellent thermal shock resistance and are reasonably ductile. They were developed by D. G. Moore and J. R. Cuthill of the enameled metals laboratory.

Cermets possess properties of both metals and ceramics. The chromium-boron-nickel types have some degree of ductility. They are resistant to thermal shock and oxidation at high temperatures, and retain their strength and hardness at high temperatures as well. In recent years, chromium-boron-nickel layers have been used extensively as hard facings for metal parts subjected to the severe conditions of erosion and oxidation. In one commonly-used process, the chromium-boron-nickel powder is flame-sprayed onto the metal part. This overlay is then fused to the base metal with an oxyacetylene torch. The resulting layer is dense and well-bonded but has several disadvantages. First, it

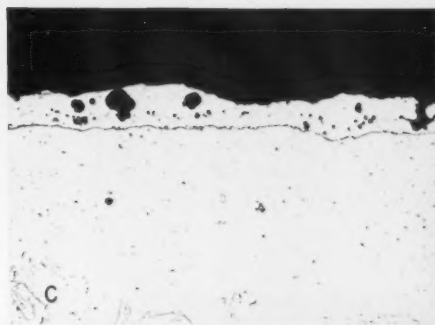
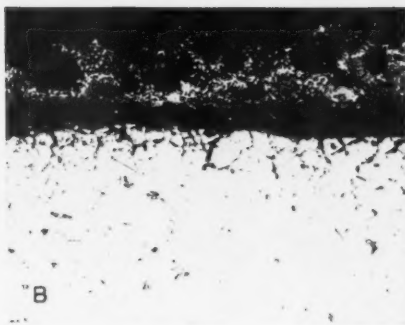
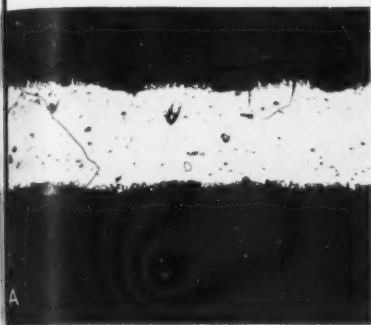
tends to be expensive for large-scale production; second, the flame-spray technique is unsuited to the coating of cylindrical parts of small inside diameter; third, the process is not feasible where thin layers—about 0.002 in.—are required. To overcome these objections, the Bureau undertook a study of other methods of applying chromium-boron-nickel cermets. So far, the use of ordinary ceramic coating procedures appears to be the most successful.

In the NBS process, a slip is prepared from a cermet powder and a ceramic frit. However, instead of flame-spraying the material, the usual ceramic coating procedure is followed and the slip is applied to the metal part at room temperature by either dipping or spraying. The part is then fired at a temperature above the melting point of both the cermet and the frit. During the firing, the ceramic phase in the coating serves as a flux to permit welding of the cermet particles without the necessity of a highly-purified, oxygen-free atmosphere. Depending on the firing temperature, the ceramic material either “sweats out” to the surface or remains as occlusions in the cermet network. At the same time the cermet particles melt to form a continu-

The Bureau's M-60 cermet coating slip is applied to a gas turbine part by spraying at room temperature. The NBS process is more readily adaptable to the coating of complex shapes than is the older flame-spraying method. This turbine part will be used experimentally to determine how much the M-60 coating increases service life under high-temperature conditions.







Micrographs of three low-alloy steel specimens after long-time heating in air at 1,500° F. for cermet coating evaluation at the Bureau. Specimen A, which was uncoated, shows only a small amount of metal remaining after 400-hr. heating. (Dark areas are oxide scale.) Oxide layer and stringers under NBS ceramic coating A-19H on specimen B show that the protection provided by the all-ceramic coating was not nearly as good as that obtained with NBS cermet coating M-60F on specimen C. Nitral etch, approx.  $\times 125$ .

ous layer that becomes brazed to the surface of the basis metal.

In one series of experiments, coatings with different ratios of frit to cermet powder were prepared and applied to various basis metals. It was discovered that the sweating out of the glassy phase to the surface of the cermet layer occurred at a firing temperature of 1,900° F for coatings which contained 5, 10, or 20 parts by weight of frit. However, a temperature of 2,100° F was required before sweating out occurred with a coating which contained 50 parts by weight of frit. In the cases where sweating out did not occur at all, the glassy phase was found to be enmeshed in a welded network of the cermet material.

Firing time had little effect on the appearance of the coatings. A coating appears the same after 16 min of firing as it does after 2 min. Firing in air gave satisfactory coatings on alloys that initially had good oxidation resistance, but a furnace atmosphere of low oxygen content was necessary to achieve satisfactory protection on such metals as molybdenum, ingot iron, and several low-alloy steels.

Neither thorough cleaning nor roughening of the metal surface prior to applying the coating was essential. Satisfactory coatings were obtained on smooth surfaces which had been only mildly degreased before applying the slip.

The coating with the best continuity of the Cr-B-Ni layer, and with the best appearance, was obtained from a mixture of 10 parts of frit, 90 parts of cermet powder,

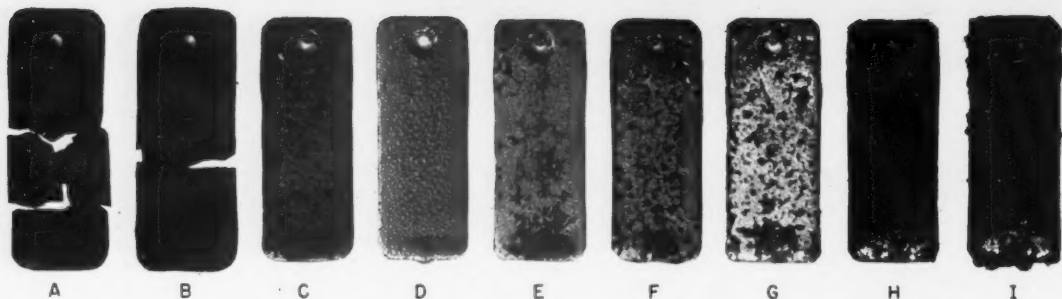
After coating with NBS cermet coating M-60, the turbine part is fired in an electric furnace at 1,975° F in an air atmosphere. During the firing the ceramic phase in the coating serves as a flux which permits welding of the cermet particles to the basis metal without the need of a highly purified, oxygen-free atmosphere. The final result is a hard, high temperature-resistant facing on the metal part.

Specimens coated with Cr-B-Ni cermets fired for 4 min. at 1,950° F in air. A contained Cr-B-Ni cermet powder only; B contained 100 parts by weight of cermet powder and 5 parts clay; C contained 90 parts cermet powder, 10 parts NBS frit 332, and 5 parts clay. True welding of cermet to give a continuous metallic-like layer occurred only in coating containing frit.



and 5 parts of clay by weight. This coating was designated M-60. With this mixture, coating thicknesses in the range of 0.004 to 0.008 in. can be achieved by single-coat application. For thicknesses greater than this range, multiple coatings are recommended. For thinner coatings, finer grinding of the slip is required. Such thin compositions have been designated M-60F.





Effect of heating in air at 1,800° F on specimens of ingot iron coated with NBS ceramic coating A-19H and cermet coating M-60. Specimen A was uncoated, B was coated with A-19H, and the remaining specimens with M-60. A, B, C, and E were heated for 24 hr. D was not subjected to heating. The other specimens were heated as follows: F, 48 hr.; G, 92 hr.; H, 192 hr.; I, 380 hr. Mottled appearance of specimens coated with M-60 is caused by non-uniform flaking of thin scale layer that formed on the cermet coating surface during the heating.

With the coarsely ground coating (M-60), the glassy phase forms at the surface of the cermet as small beads when fired according to the recommended procedure. These beads tend to flake off on cooling, but light sand-blasting or treating in molten alkali will result in a specimen of more acceptable appearance. On the other hand, when the finely-ground coating (M-60F) is used, and application thicknesses are less than 0.003 in., the glassy phase that comes to the surface forms a uniform glaze that adheres to the underlying cermet layer. In this case, the specimen appears to be ceramic coated.

The possible use of the M-60F coating for protecting low-alloy steels against oxidation at 1,500° F was also investigated. Uncoated specimens scaled rapidly, whereas the M-60F coating prevented any loss in effective thickness for the maximum test period of 800 hr. However, spectrochemical analysis shows that boron diffuses into the alloy structure during the heat treatments. In one test, the boron content of a low-alloy steel reached 0.028 percent by weight after the 800-hr treatment at 1,500° F.

Batch composition and milling instructions for NBS cermet coatings M-60 and M-60F

Batch composition		
Ingredient		Parts by weight
Cr-B-Ni powder <sup>a</sup>		18
Frit 332 powder <sup>b</sup>		2
Enameling clay		1
Water		5
Milling instructions		
	M-60	M-60F
Milling time <sup>c</sup>	1 hr	17 hr
Specific gravity of slip for optimum workability	3.25	2.60

<sup>a</sup> Chromium-boron-nickel cermet powder sized to pass a U. S. Standard 140-mesh sieve and pretreated for removal of lubricant.

<sup>b</sup> Prepared by ball-milling NBS frit 332 in a dry mill until it is sufficiently pulverized to pass a U. S. Standard 40-mesh sieve.

<sup>c</sup> In 1-gal ball mill with zircon balls using 1,575 g of dry-coating ingredients and 375 g of water.

## Portable Standard Cell Comparator

THE BUREAU has devised a convenient, inexpensive method for maintaining the standard volt to 0.002 percent in the laboratory. Developed by A. W. Spinks and F. L. Hermach of the NBS electrical instruments laboratory, the method uses apparatus that is simple to operate and requires little maintenance. The equipment consists essentially of four saturated standard cells, a temperature-controlled airbath for the cells, and a thermofree potentiometer for intercomparing them.

To an increasing extent, scientific and industrial laboratories are using small groups of standard cells as stable, accurate sources of reference voltage. Normally, one or more cells in a group are certified periodically at a standardizing laboratory such as NBS.

Between certifications all cells are intercompared frequently to detect relative drifts or erratic changes in emf.

The saturated standard cells (containing an excess of cadmium sulphate) that are usually employed for this purpose are much more stable than the common unsaturated cells. However, saturated cells have a negative temperature coefficient of about 50 microvolts per degree C at room temperature, so that some form of constant temperature enclosure is necessary. A temperature-regulated oil bath, containing continuously stirred refined mineral oil, constitutes the usual laboratory equipment for maintaining saturated cells at a constant temperature. However, an oil bath is bulky, expensive, and nonportable and may involve consider-



able maintenance. The NBS airbath device, on the other hand, is relatively lightweight and inexpensive and is sufficiently rugged for general laboratory use. It was originally designed by H. F. Stimson of the Bureau's temperature measurements laboratory.

The standard cell container of the comparator is made of two cast aluminum boxes, one inside the other. A thermal insulating layer of balsa wood separates the boxes, and another layer surrounds the outer box. Four standard cells are placed in the inner box which, in turn, is surrounded by electrical heating coils. Continuous power is required from a 60-cycle, 120-volt line to maintain the cells at 34° C. The power is supplied through a stepdown transformer and is controlled by a thermoregulator.

Failure of the power supply (i. e., loss of temperature control) is indicated by a loss in time as shown by a synchronous clock connected to the input power line in the regulator circuit. A mercury-in-glass thermometer, with its bulb in the outer box, indicates the operating temperature. The scale of the thermometer is read with a low-power microscope.

The built-in Lindebeck potentiometer is used to compare the emf's of the cells. This potentiometer provides known voltage differences by utilizing the voltage drop produced in a special four-terminal shunt by a measured current. This adjustable current is measured with a 2-ma milliammeter.

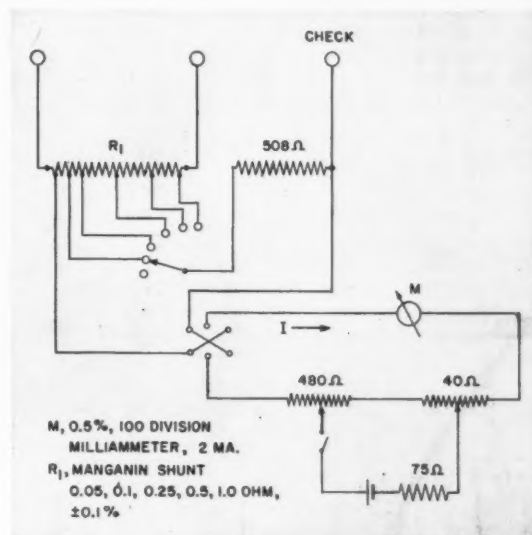
The special four-terminal multiple-range shunt consists of an annealed manganin strip to which are silver-

Portable equipment for intercomparing standard cells to an accuracy of 0.002 percent includes a precision potentiometer with an electrically heated temperature-controlled enclosure for the cells. Low-power microscope at top is used to read thermometer which indicates temperature of cell enclosure.

soldered long copper leads to provide appropriate potential taps. The resistor is thermally shielded by a copper box which surrounds it. Each resistor section is adjusted to the desired value to better than 0.1 percent.

To perform an intercomparison, the standard cell of known voltage and the cell of unknown voltage are connected in series, emf's opposing, and the small difference in their potentials is measured with the potentiometer. The negative terminals of the two cells are connected to each other, and the positive terminal of the unknown cell is connected to one post of the potentiometer. The positive terminal of the known cell is connected through a galvanometer and a switch to a second post of the potentiometer. When the voltages are balanced, the difference in the emf's of the two cells is equal to the product of the milliammeter reading and the resistance value of the shunt for the taps used. A reversing switch indicates by its position which cell has the higher emf. By applying this difference to the value for the known cell, the emf of the unknown cell is determined.

In order to insure the most reliable accuracy, it is essential that the standard cell comparator be maintained continuously at a constant temperature. The thermoregulator used in the NBS comparator has an inherent sensitivity of  $\pm 0.02$  deg C. Tests have shown that the temperature of the cells does not vary by more than 0.01 deg C. Thus, changes in emf arising from temperature variations of the cells in the comparator should be less than 0.5 microvolt.



Wiring diagram of potentiometer circuit in NBS standard cell comparator.

# A Rotating Reading Head for Magnetic Tape and Wire

**A** READING HEAD that makes possible the close examination of a short section of magnetic tape or wire is now being used at the Bureau to locate and investigate faults in magnetic recording media. Developed by J. R. Sorrells of the NBS data processing systems laboratory, this instrument makes use of a reading head mounted on a rapidly rotating drum so that the head is in contact with the tape for a part of each revolution. Since the tape is held stationary, the head reads exactly the same set of signals once each revolution, and the playback can be displayed continuously on an oscilloscope and observed as long as desired.

In addition to providing a means for closely examining the playback signals from a specific portion of the tape, the reader can be used to scan through and edit a complete tape. The observer can easily locate any defective signals along the entire length of the recorded tape. Interchangeable parts provide a means for examining several different sizes of magnetic tape or wire.

In the design and development of magnetic tape and wire equipment for external pulse storage in electronic digital computer systems, one of the primary considerations has been reliability of operation. An important factor in magnetic storage is the condition of the tape surface itself. Errors in operation can be caused by any of several types of tape faults such as "holes" and raised spots in the magnetic surface, or creases in the tape. Very often the loss of several pulses or the gain of a single pulse may be caused by a flaw that is too small to be visible to the unaided eye. Conventional means of tape reading are not suitable for locating errors, since in the usual tape transport mechanism the tape is moved continuously past a stationary head. In investigating tape for faults it is desirable to read a small specific portion of the tape over and over again at a rapid rate, and to display on an oscilloscope a steady, clear picture of the playback signals. The rotating head reading device developed at NBS provides such a repetitive method for examining tape. Once faults are located, they can be removed or else avoided in the future, thus increasing the reliability of the tape.

For convenience, the tape reader is mounted on a vertical panel. Near the top upper corners are the shafts on which the tape reels are mounted. Although tape must be reeled manually on the NBS model, a motor drive or stepping mechanism could easily be attached. The idler shafts are friction loaded to maintain the proper tension on the tape for reading as the tape is reeled along.



Close examination of a short section of magnetic tape or wire is possible with this device. It utilizes a drum (center of panel) with a reading head which is rotated repetitively over a short segment of tape. The same set of signals are read repetitively and may be displayed continuously on an oscilloscope. Inserts show rotating heads for tape (lower left) and wire (upper right).

At the lower center of the panel is the rotating drum on which the reading head is mounted. The drum is  $2\frac{7}{8}$  in. in diameter and rotates at 10 rps. This is equivalent to a tape speed of 90 in./sec. The drum is made in two sections. The reading head is mounted on one section and has a groove or track machined around the periphery in which the tape or wire rides. This section is easily removed, and other similar sections may be substituted for different sizes of tape or wire.

The section of the drum nearest the panel is fastened to the drive shaft and is not ordinarily removed. This section is cup-shaped with the open side toward the panel. A photoelectric cell is mounted on the panel, inside the cup-shaped section, close to the periphery of the drum. A small aperture in the drum wall makes it possible to focus an externally mounted lamp on the photocell when the drum is in the proper position.

Each time the hole in the drum wall passes the light source, the light strikes the photocell and causes a small output pulse from the cell. The pulse is applied to a cathode follower which in turn triggers the oscilloscope sweep. The aperture in the drum wall is so located that the trigger pulse occurs just before the reading head makes contact with the tape, so that the oscilloscope sweep always begins slightly before the first pulse is sensed by the head. It is this timing system that provides a steady picture of the repeated playback signals on the oscilloscope screen.

Since the reading head is mounted on a revolving drum, electrical connections must be made through slip-rings and brushes. A specialized slipring assembly using commercial brushes was designed and constructed for this purpose. The three rings are made



Interior view of the mechanism for the rotating head magnetic tape or wire reader. Reading heads are used at NBS to locate and investigate faults in magnetic media.

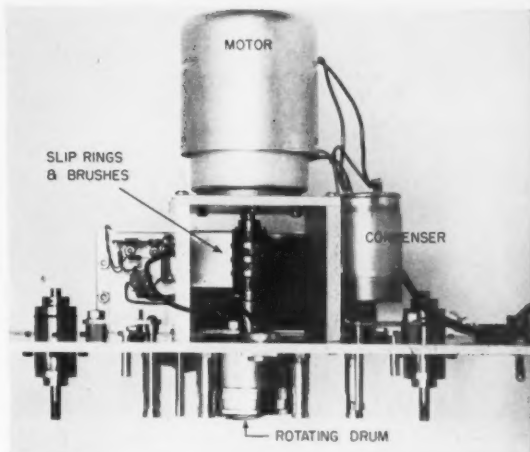
of electrodeposited silver on a premachined bakelite cylinder; the silver is further machined for good contact surfaces. The cylinder is mounted between the drum and the driving motor, and a two-wire shielded cable runs from the sliprings through the shaft to the reading head.

On the front of the panel are four grooved studs that guide the tape as it enters and leaves the reels and the rotating drum. Rubber shoes pressing firmly against the studs prevent the tape from creeping while being scanned. Interchangeable studs are provided for different sizes of tape and wire.

Trials of the equipment in the laboratory prove its usefulness in locating tape flaws and in reading recorded pulses. The playback signal on the oscilloscope screen shifts such a small amount that photographs taken with an exposure time of 15 sec reveal no evidence of blurring. Moreover, there is no noticeable noise from the slipping and brush assembly.

By expanding the oscilloscope sweep, it is possible to read a computer word simply by recognizing the value of each recorded digital pulse. The rotating head has been used in this way to compare information on a magnetic wire with the paper tape from which it was recorded. This method is used to determine whether the transcribing equipment is causing trouble or the wire is at fault.

In addition to being an effective and useful means of investigating magnetic recording phenomena, the rotating-head type of the reader could also be used as a means for transcribing information directly from the keyboard to the magnetic tape. It would be most convenient to use a multichannel tape together with some



provision for advancing it in short, precise steps. Each time a key on the keyboard is pressed, the corresponding character in coded form is set up in an electronic register. Then at a specific point of the rotating drum revolution, the contents are recorded on the tape in parallel form. On the next revolution of the drum, the character just recorded is compared with the character stored in the register. If the two agree, the tape advances a small distance, and the next key can be depressed to begin the next record-read-check cycle. If the two characters do not agree, the tape advance mechanism is locked out, an error indicator flashes, and the operator can either try to record again or find where the difficulty is. In this way, an operator could transcribe his problem directly from his manuscript to a magnetic tape, which then could be read directly into the computer.

## Nature of Stark Rubber

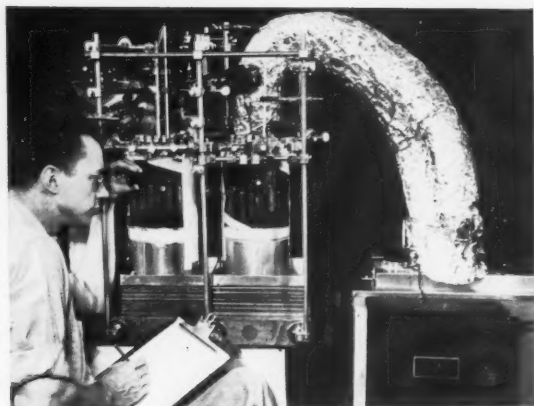
**T**HE PHYSICAL PROPERTIES of polymers and their ultimate end uses as rubbers, plastics, or fibers depend to a marked extent on the presence or absence of crystallinity and on the melting temperature. Many plastics and fibers would be rubbers if they were not crystalline at ordinary temperatures; actually these materials do become rubberlike above their melting temperatures. On the other hand, many rubbers owe their strength to the development of crystallinity on stretching. Yet rubbers that develop appreciable crystallinity on cooling tend to lose their elasticity and extensibility at low temperatures and are thus unsuited for use at very high altitudes or in arctic regions.

To obtain information which may serve as a guide in the preparation of new types of polymers, the Bureau is conducting a program of basic research on the crystallization properties of polymers and their correlation with molecular structure. A recently com-

pleted phase of the program has dealt with the nature of stark rubber.<sup>1</sup> This work was carried out by D. E. Roberts and Leo Mandelkern of the NBS rubber laboratory and was partially supported by the Office of Naval Research.

The term "stark" is applied to those occasional specimens of natural rubber which are found to be hard and inelastic after prolonged storage in temperate climates. The hardness and inextensibility are due to the development of appreciable crystallinity during storage. Perhaps the most unusual property of stark rubber is its melting behavior. The melting point of stark rubber has been reported to be considerably higher than that of ordinary natural rubber.<sup>2</sup> However, once melted, stark rubber thereafter melts at the lower temperature and otherwise behaves like ordinary natural rubber.

The anomalous melting behavior of stark rubber has



Apparatus used at NBS to observe volume-temperature changes in stark rubber. Samples are contained in small volume dilatometers held at constant temperature within the two glass jars. Changes in sample volume are observed as the displacement of a mercury meniscus within a capillary tube connected to the sample chamber of the dilatometer. Refrigeration unit at right is part of the temperature-control system.

long been an obstacle to more complete understanding of the thermodynamics of polymer crystallization. To find out how stark rubber is formed and the reasons for its high melting temperature, the Bureau investigated the melting behavior and X-ray diffraction patterns of four stark rubber samples from widely different sources.

In studying the melting behavior of the stark rubber samples, the Bureau used volume dilatometers of conventional design to observe the variation of the specific volume with temperature. These observations confirmed the higher melting temperature of stark rubber. The samples studied had melting temperatures in the range from  $39^{\circ}$  to  $46^{\circ}$  C, as compared to a melting point of  $28^{\circ}$  for natural rubber crystallized by cooling only.

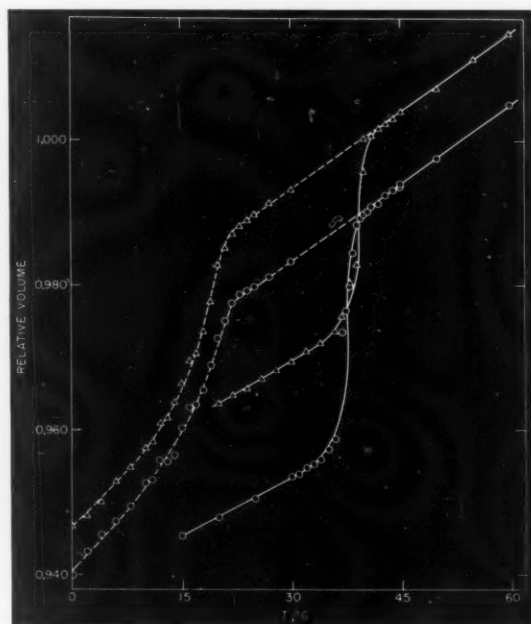
The melting behavior of the stark rubber was also quite different in other ways from that of ordinary natural rubber and other polymers. In the temperature interval from about  $35^{\circ}$  to the melting temperature, the specific volume increased very slowly under isothermal conditions. Yet the total volume change at a given temperature was appreciable. This made it necessary to raise the temperature of the samples very gradually. Thus the total heating process for a typical determination of the melting temperature required from 50 to 100 days.

X-ray diffraction patterns obtained from the stark rubber samples at room temperature gave a rather clear indication of the basis for the observed crystallization behavior. Though the diffraction haloes corresponded to the spacings observed in natural rubber that has crystallized either by cooling or stretching, the intensities of the diffracted X-rays around the circumference of the rings were not uniform. This result indicates a preferred orientation of the crystallites as compared to the random orientation observed in systems where crystallization is induced solely by cooling. Because the crystallites are oriented, the amorphous

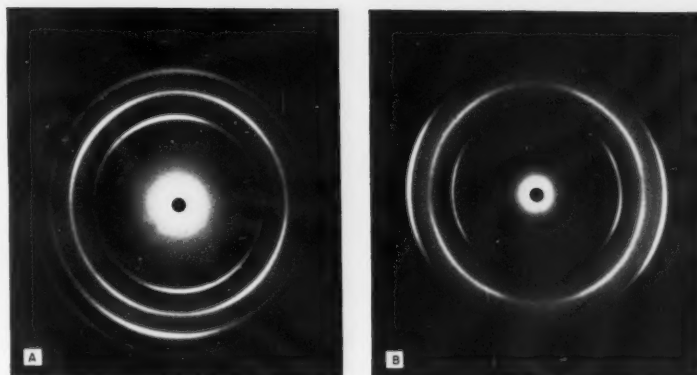
Unusual melting behavior of stark rubber is shown by plotting relative volume against temperature for two samples studied. Solid lines show melting during the first heating. Dashed lines show melting when the same samples were first melted and then crystallized at  $0^{\circ}$  C.

regions that connect them must also be oriented to some extent. In such an oriented system there are two possible explanations for the higher melting temperature. If the orientation is maintained on fusion, the melting temperature will be raised because less entropy is gained on melting relative to the unoriented system. Alternatively, at an appropriate temperature the oriented amorphous regions can rearrange themselves to their new probable configurations, and the crystallites will become unstable and melt. The latter behavior is a more likely possibility in the case of stark rubber, since normal crystallization behavior is observed after the initial melting.

The orientation in stark rubber is probably due to the original plantation processing. After coagulation of the latex, the amorphous rubber is rolled into sheets, and the sheets are stacked one upon another. The bale of rubber is then subjected for a time to a rather large simple compressional stress. It is likely that this stress can cause the amorphous segments to be preferentially oriented, and because of the high viscosity of natural rubber, this orientation can persist for long periods of time at the usual storage temperatures when the external stresses are removed. Orientation of the chain segments, in turn, facilitates crystallization at temperatures where crystallization is prohibitively slow for undeformed rubber.



Typical X-ray diffraction patterns obtained with a stark rubber specimen at room temperature. Preferred orientation of crystallites in stark rubber is indicated by the nonuniformity of some of the rings. A, Naturally occurring sample; B, sample prepared in the laboratory.



In principle, other crystalline polymers could display this same melting behavior. In stark rubber the high viscosity of the amorphous material allows the orientation of the chain segments to be maintained for relatively long periods of time in the temperature range of interest. The lower limits of this temperature range are set by the equilibrium melting temperature of natural rubber. As long as the equilibrium melting temperature is sufficiently low, as in the case of natural rubber, for the rate of amorphous rearrangement to be slow, this phenomenon should be observed.

As a result of the Bureau's studies, it is possible to prepare stark rubber in the laboratory under controlled

conditions. The essential requirement is orientation during the crystallization. Conditions should be provided such that crystallization occurs while the rubber is being deformed, and that when the external stresses causing the deformation are removed, the orientation will persist at temperatures above the equilibrium melting temperature of natural rubber.

<sup>1</sup> For further details, see Nature of stark rubber, by Donald E. Roberts and Leo Mandelkern, *J. Research NBS* **54**, 167 (1955) RP2578.

<sup>2</sup> Thermodynamics of crystallization in high polymers: Natural rubber, by Donald E. Roberts and Leo Mandelkern, *J. Am. Chem. Soc.* **77**, 781 (1955).

## New Motion Picture

### Dental Materials: Specification and Certification

NBS in cooperation with the American Dental Association has produced a 16 mm, sound and color 21 min film intended to aid the dentist in a better understanding of the Certification Program of the American Dental Association.

This film describes the Specification and Certification Program of the American Dental Association's Research Fellowship at NBS. The technical procedure used in the development of a specification is presented, using silicate cement as an example.

The film is designed for showing to professional groups, at clinics or scientific sessions and to general practitioners of dentistry. It is also of value as a teaching aid in dental schools for demonstrating the methods by which a dental material is tested and certified as complying with an official ADA specification.

For information about the loan or sale of this film, write to the Office of Technical Information, National Bureau of Standards, Washington 25, D. C.

## Publications of the National Bureau of Standards

Journal of Research of the National Bureau of Standards, volume **55**, No. 1, July 1955 (RP2599 to RP2604 incl.). Annual subscription \$4.00.

Technical News Bulletin, volume **39**, No. 7, July 1955. 10 cents. Annual subscription \$1.00.

CRPL-131. Basic Radio Propagation Predictions for October 1955. Three months in advance. Issued July 1955. 10 cents. Annual subscription \$1.00.

### Research Papers

*Journal of Research*, volume **55**, number 1, July 1955. Single copies of the *Journal* vary in price. Single copies of *Re-*

*search Papers* appearing in the *Journal* are not available for sale. The Superintendent of Documents, U. S. Government Printing Office, Washington 25, D. C., will reprint 100 or more copies of a Research Paper. Request for the purchase price should be mailed promptly to that office.

RP2599. Some properties of a glass used in paper manufacture. Martin J. O'Leary and Donald Hubbard.

RP2600. Adsorption of nitrogen on carbon absorbents at low pressures between 69° and 90° K. Juan de Dios Lopez-Gonzalez, Frank G. Carpenter, and Victor R. Deitz.

RP2601. Stress-strain relationships in yarns subjected to rapid impact loading: 3. Effect of wave propagation. Jack C. Smith, Frank L. McCrackin, and Herbert F. Schiefer.



# TECHNICAL NEWS BULLETIN

U. S. DEPARTMENT OF COMMERCE

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NATIONAL BUREAU OF STANDARDS

A. V. ASTIN, *Director*

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## NBS Publications (continued)

RP2602. Thermal expansion and phase transformations of low-expanding cobalt-iron-chromium alloys. Peter Hidnert and Richard K. Kirby.

RP2603. Vapor pressures of the methanes. George T. Armstrong, F. G. Brickwedde, and R. B. Scott.

RP2604. Data on the atomic form factor: Computation and survey. Ann T. Nelms and Irwin Oppenheim.

### Circulars

C560. Leather research and technology at the National Bureau of Standards. Everett L. Wallace. 15 cents.

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